

**DEVELOPMENT OF ORGANIC CONJUGATED POLYMER  
MATERIALS FOR ENERGY HARVESTING  
THESIS**

**SUBMITTED TO LINGAYA'S VIDYAPEETH , FARIDABAD IN PARTIAL  
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**by**

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**DEPARTMENT OF BASIC SCIENCES**

### **Declaration**

I declare that the thesis on “**Development of organic conjugated polymer materials for energy harvesting**” has been composed by myself and that the work has been submitted only for Partial Fulfillment of the requirements for the degree of Masters of Science in Physics. I have adequately cited and referenced the original sources. I also declare that I have adhered to all principles of academic honesty and integrity and have not misrepresented or fabricated or falsified any idea/data/fact/source in any submission. I understand that any violation of the above will be cause for disciplinary action by the Institute and can also evoke to penal action from the sources which have thus not been properly cited or from whom proper permission has not been taken when needed.

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**Date**

*Shivangi sharma*

**Shivangi Sharma**

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This is certify that M.Sc. (Physics) project report entitled “**Development of organic conjugated polymer materials for energy harvesting**” submitted by “**SHIVANGI SHARMA**” to Lingaya’s Vidyapeeth is a record of Bonafide work carried by her under our supervision. The project report submission in partial fulfilment of the requirement for the award M.Sc. in Physics in the department of Physics in Lingaya’s Vidyapeeth (Haryana).

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## Abstract

In recent years alternative renewable energies obtained by solar cells have attracted much attention due to exhaustion of other conventional energy resources especially fossil based fuels and due to global warming they caused [1]. Thus the synthesis of novel conducting polymers and study of their physical properties has been of prime importance. Aqueous electrochemical process in an environmentally friendly and efficient technique used to process conducting polymer. It is widely preferred because of its simplicity and it can be used as a one step method to form polymer. It allows efficient control of the physiochemical properties of the coatings and it can also be easily scaled up for large scale production [2-4]. Conductive polymers had been the topic of the large number of investigations during last decades because of their unique properties such as mechanical strength, electrical conductivity, corrosion, stability and possibility of both oxidative and electrochemical synthesis. The obtained polyaniline compound in powdered form is then converted to make pellet and these pellets are used further for the characterisations like X-Ray Diffraction (XRD), Fourier Transformed Infrared analysis (FTIR) & at low temperature conductivity. Hence PANI is useful in wide area of application: such as solar energy conversion, rechargeable batteries, electrochromic displays, electrochemical sensors, capacitors and active corrosion.

In the present study, the monomer of aniline has been prepared from the raw aniline by the chemical polymerization of aniline was carried out in the presence of aqueous sulphuric acid solution with the chemical oxidative method, to get the powdered form of polyaniline which has been further used with different compositions to make pellets with other compounds like GO and PVDF in different ratios with their different compositions. These pellets are further used to characterise to validate the synthesis of polyaniline, to confirm the obtained compound is polyaniline or not. The characterisation of polyaniline is done by using three well known patterns from XRD, FTIR and low temperature conductivity methods. To be concluded the size, shape and structural units of polyaniline the XRD method is used, for the broad spectrum it is obtained through FTIR method and also for variations of conductivity with temperature changes it is obtained by using low temperature conductivity method.

The graphs plotted between Intensity and diffraction angle in XRD method is used to identify the peaks which further confirms the size, shape and structural details of polyaniline. For the polyaniline/GO composite. The graphs plotted between transmittance and wave number in FTIR method is used to obtain an infrared spectrum of absorption

or emission of a solid, liquid or gas. The graphs plotted between conductivity and temperature in low temperature conductivity method is used to determine the variation in conductivity. It is concluded from the graph that the conductivity of the resulting composite is  $1.401 \times 10^{-8} \text{ S cm}^{-1}$  at room temperature, which is higher than that of pure polyaniline.

The main aim of this research work is to develop energy efficient materials for the present need by comparing their electrical conductivity properties. In this paper, we report the synthesis, characterization and conductivity studies of polyaniline prepared by electrochemical and chemical oxidation methods. Further, their electrical conductivity was compared with electrochemical, synthesis of PANI doped with  $\text{H}_2\text{SO}_4$  at various voltages.

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## Organization of Thesis

The work presented in the dissertation has been divided in to five chapters as follows:

**Chapter 1** deals with the introduction part of the present study and its background.

**Chapter 2** deals with the experimental part having details of material used, measurement to be concluded from the research, synthesis of the polymer polyaniline, how the polythiophene has been formed and the pallet formation from the compound.

**Chapter 3** deals with the characterization of the compound using available techniques i.e. Fourier transformed infrared spectroscopy, x-ray diffraction and low temperature conductivity.

**Chapter 4** deals with the results and discussion concluded from the research carried out.

**Chapter 5** deals with the advantages and disadvantages of the conducting polymer.

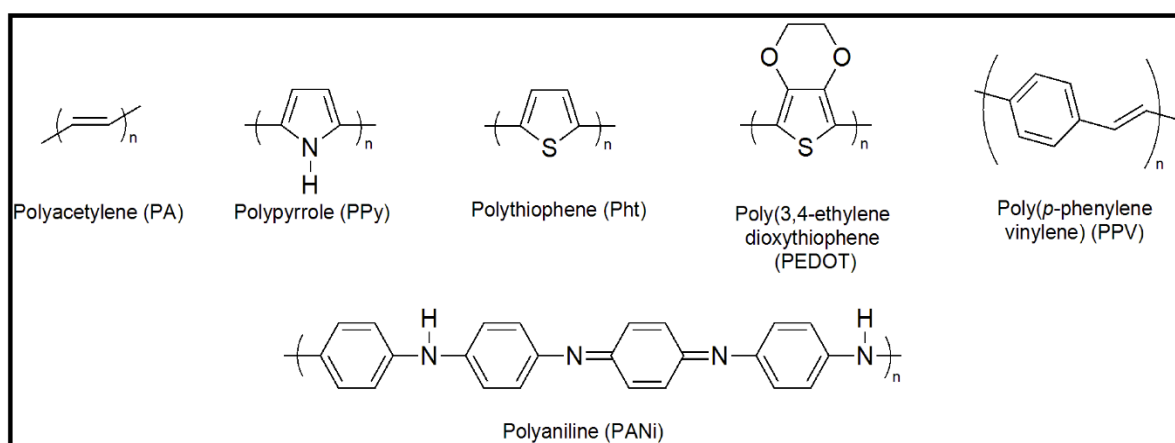
# Chapter - 1

## Chapter – 1: Introduction

### 1.1 Conjugated polymers

Conjugated polymers are organic macromolecules that are characterized by a backbone chain of alternating double-bonds and single-bonds. Their overlapping p-orbitals create a system of delocalised  $\pi$ -electrons, which can result in interesting and useful optical and electronic properties. Till the early 19th century all the developed polymers were non-conducting. In the year 1976 Alan J. Heeger and his colleagues Alan G. Macdiarmid and Shirakawa discovered the conductive properties of polyacetylene which was comparable to that of a metal. Oxidising the PA using iodine vapour film increases the conductivity of PA greatly ( $\approx 3000$  S/cm) over the undoped material. The aftereffects of this discovery led to discovery of many more conducting polymers like Polypyrrole, polythiophene, poly(p-phenylene), polyaniline and their derivatives. The key property of conducting polymers was the presence of conjugation i.e. the presence of alternative single and double bond. The property of conjugation is necessary but not enough property to make the polymer conducting, it is where dopant plays in generating charge carriers along the polymer chain thereby giving rise to electrical conductivity. A hole is created when the conjugated polymer is oxidised in polymer chain, this hole allows electron from neighbouring atom to jump due to conjugation thereby creating new hole, the movement of hole along the chain of give rise to the current. Some of the conducting polymers are shown in that figure 1.1.

Numerous studies have been done on various conductive polymers to enhance their conductivity and utilize their applicability in various technological areas such as field effect transistors, light diode, organic thermoelectric etc. [2]



**Figure 1.1** Some of the examples of conjugated polymers(Wikipedia)

### **1.1.1 Principle of electrical conduction- mechanism and doping**

Conducting polymers are intrinsic semiconductors in nature. This is due to presence of conjugation of the backbone chain. The overlap of  $\pi$  electrons cloud forms a delocalized molecular wave function. This delocalization assists in transfer of charge and hence contributes towards conduction. The role of dopant is to add or remove an electron from the chain. For example, in  $I_2$  doped Polyaniline  $I_2$  extracts an electron from the chain and forms  $I_3^-$  ion, this is an example of oxidative doping, the vacancy created leads to the formation of a radical polaron. The charge is carried along the chain however the counterion ( $I_3^-$ ) associated with the positive charge is entrapped and is immobile. High concentration of polaron is required so that it can move in the field of nearby polaron. Hence higher doping of counterion is required for a highly conducting polymeric species. If the second electron is removed a fresh polaron is created and if the electron from the unpaired electrons of the existing polaron a bipolaron is formed, which moves in a pair. Other type of species formed is solitons, it is like a defect in the chain which can move along the conjugated backbone of the chain but itself doesn't carry a charge.

The formation of the above-mentioned species affects the energy level of the polymers. To form the negative polaron an electron from the donating species is added in the upper shell whereas to form the positive polaron the electron from the lowest polaron level is taken and a hole is created. The energy difference between the polaron level and the closest band edge is dependent on the conjugation length. and thus, creating the species like polaron, bipolaron and solitons. The doping process is more of a redox reaction where reduction or oxidation of conjugated chain form an anion or cation and which is attached to the counterion by the coulombic force.[8]

### **1.1.2 Synthesis of conducting polymers**

Conductive polymers can be prepared by various methods like Chemical oxidative polymerization, metal-catalysed polymerization, photochemical polymerization, solid-state polymerization, plasma polymerization, interfacial polymerization and electrochemical polymerization. Here we discuss chemical oxidative polymerization which is mainly employed during the project.

In chemical oxidative polymerization method, the monomer is first oxidised to form a radical cation species using a suitable oxidising method. This radical cation further reacts to form a dimer and subsequently the dimer is oxidised again and reaction result in a polymerization of

the monomer. This method is widely adapted among researchers to form conducting polymers. Here since the kinetics is involved, the temperature of the reaction plays the major role. Polypyrrole and its composite are prepared using this method in the current project.

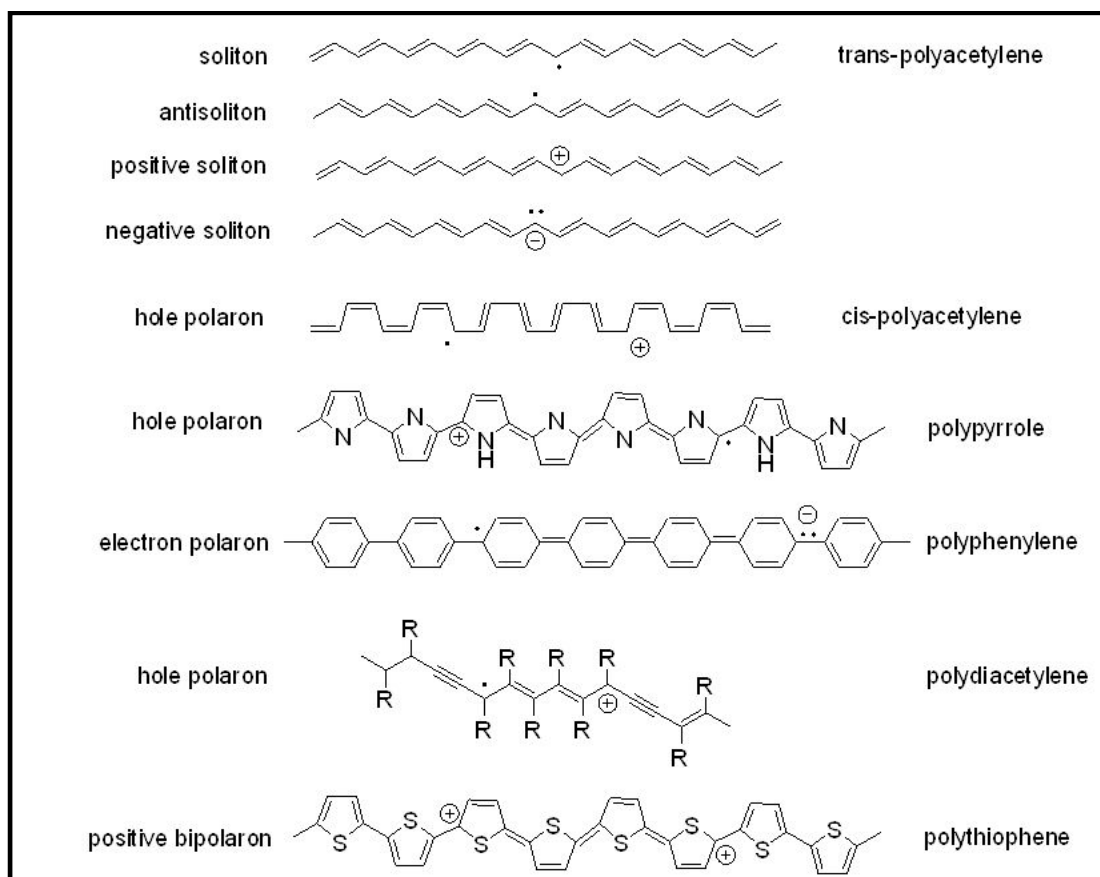
A polymer material containing a long chain of molecular structures is first and foremost an insulator. The idea that polymers or plastics could conduct electricity is considered absurd. Their wide application as an insulating material is the reason they are studied and developed in the first place. Truth be told, these materials are regularly utilized for encompassing copper wires and assembling the external designs of electrical machines that keep people from coming in direct contact with power. Around thirty years prior, researchers found that a sort of formed polymer called 'polyacetylene' could turn out to be profoundly electrically conductive in the wake of going through a primary adjustment measure called Doping.

The polymer is known as a 'conjugated polymer' in light of the rotating single and twofold bonds in the polymer chain. Because of the exceptional formation in their chains, it empowers the electrons to de-restrict all through the entire framework and along these lines, numerous molecules might share them. The de-limited electrons might move around the entire framework and become the charge transporters to make them conductive. This polymer can be changed into a directing structure when electrons are eliminated from the spine bringing about cations or added to the spine bringing about anions. Anions and cations go about as charge transporters, bouncing starting with one site then onto the next affected by an electrical field, hence expanding conductivity. Be that as it may, the Conjugated polymers are not conductive, since they are covalently fortified and don't contain valence bands like unadulterated metal does. It is all around concurred that the doping system is a successful technique to create conducting polymers.

Doping permits electrons to stream because of the development of conduction groups. As doping happens, the electrons in the formed framework, which are approximately bound, can hop around the polymer chain. The electric flow will be created when the electrons are moving along the polymer chains. A few instances of conjugated polymers are polyacetylene, polypyrrole, polyaniline, and polythiophene. Later on, leading polymers or natural metals might supplant inorganic metal in a few basic regions.

Certain parts of the inorganic metals, for example, not being harmless to the ecosystem and having a high harmfulness, are the motivation behind why these natural metals have likely advantages as substitutes. The likely utilizations of these natural metals incorporate

consumption security, radars, batteries, sensors, just as electrochromic cells. Much examination will be required before the applications might turn into a reality, since formed directing polymers actually have a few disadvantages, for example, not being water solvent, having poor mechanical strength and not being biodegradable. Only time will tell us when the impact of these novel polymers will be as large as their brother's, the insulating polymer.[3]



**Figure 1.2:** The dopant plays an important role in adding and removing the electron(Wikipedia)

### 1.1.3 History

Before the disclosure of its conductive properties during the 1960s, polyaniline was considered and applied as an assortment of shaded materials and colours. The historical backdrop of the disclosure and starting points of polyaniline are given starting the underlying oxidation of aniline by F. Ferdinand Runge in 1834 and closing with the principal electrochemical oxidation of aniline by Henry Lethe by in 1862. All the while, the reports of aniline oxidation items somewhere in the range of 1834 and 1862 are assessed and examined considering current information, featuring the different recorded commitments to the current field of conjugated polymers. Finally, an initial argument for polyaniline as the first synthetic organic polymer is presented.



## 1.2 Polyaniline

Polyaniline is probably the eldest known electro conducting polymer, since it was used for textile colouring one century ago. The incredible interest in exploration of polyaniline is associated with revelation of its conductivity as emeraldine salt and presence of various oxidation forms

## 1.3 Different oxidation states of polyaniline

Unlike other known electroconducting polymers, polyaniline can exist, depending on degree of oxidation, in different forms, known as: leucoemeraldine, emeraldine and pernigraniline. Leucoemeraldine, or example leucoemeraldine base, alludes to completely diminished structure; emeraldine, for example emeraldine base, is half-oxidized, while pernigraniline, for example pernigraniline base, is totally oxidized type of polyaniline. The solitary leading type of polyaniline is emeraldine salt, acquired by doping or protonation of emeraldine base. The novel element of referenced polyaniline structures is simplicity of its shared transformations by both substance and electrochemical responses as it very well may be seen. Apart from the progressions in oxidation levels, every one of the advances among polyaniline structures are showed by shading and conductivity changes. The directing protonated emeraldine as green emeraldine salt, acquired as a result of electrochemical polymerization of aniline in acidic electrolytes, can be effectively changed by additional oxidation to completely oxidized dull blue pernigraniline salt, which can be treated by soluble base to shape violet pernigraniline. Emeraldine salt can likewise be decreased to straightforward leucoemeraldine or can be changed by soluble base to blue non directing emeraldine. two blue types of polyaniline, pernigraniline salt and emeraldine have various shades of blue. Both decrease of emeraldine salt to leucoemeraldine and oxidation to pernigraniline states are trailed by decline in conductivity.

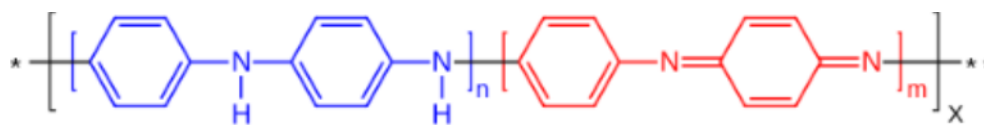
Polymerized from the reasonable aniline, polyaniline can glorify oxidation states:

- leucoemeraldine – white/clear and dull ( $C_6H_4NH$ )<sub>n</sub>
- emeraldine green for the emeraldine salt, blue for the emeraldine base ( $[C_6H_4NH]_2[C_6H_4N]_2$ )<sub>n</sub>
- (per)nigraniline – blue/violet ( $C_6H_4N$ )<sub>n</sub>

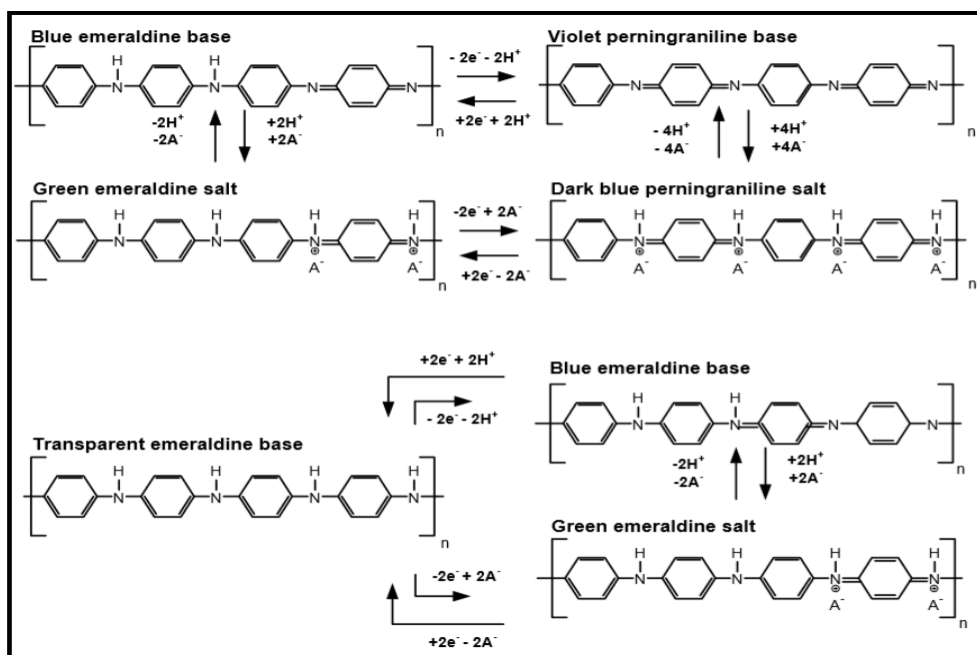
In the figure, x equivalents a large portion of the level of polymerization (DP). Leucoemeraldine with  $n = 1$ ,  $m = 0$  is the completely decreased state. Pernigraniline is the completely oxidized state ( $n = 0$ ,  $m = 1$ ) with imine connects rather than amine joins.

Studies have shown that most types of polyaniline are one of the three states or actual combinations of these parts. The emeraldine ( $n = m = 0.5$ ) type of polyaniline, frequently alluded to as emeraldine base (EB), is unbiased, whenever doped (protonated) it is called emeraldine salt (ES), with the imine nitrogen's protonated by a corrosive. Protonation assists with delocalizing the generally caught diiminoquinone-diaminobenzene state. Emeraldine base is viewed as the most helpful type of polyaniline because of its high soundness at room temperature and the way that, after doping with corrosive, the subsequent emeraldine salt type of polyaniline is exceptionally electrically leading. Leucoemeraldine and pernigraniline are poor conductors, even when doped with an acid.

The shading change related with polyaniline in various oxidation states can be utilized in sensors and electrochromic gadgets. Polyaniline sensors ordinarily abuse changes in electrical conductivity between the distinctive oxidation states or doping levels. Treatment of emeraldine with acids expands the electrical conductivity by up to ten significant degrees. Undoped polyaniline has a conductivity of  $6.28 \times 10^{-9}$  S/m, whereas conductivities of  $4.60 \times 10^{-5}$  S/m can be achieved by doping to 4% HBr. The same material can be prepared by oxidation of leucoemeraldine. [15]



**Figure 1.3:** Structures of polyaniline ( $n+m = 1$ ,  $x$  = half degree of polymerization).



**Figure. 1.4:** Different forms of polyaniline

## 1.4 Conductivity of Polyaniline

The mechanism of polyaniline conductivity differs from other electro-conducting polymers, because nitrogen atom is involved in the formation of radical cation, unlike most of the electro-conducting polymers whose radical cation is formed at carbon. Then again, nitrogen is likewise associated with the formed twofold bonds framework. Hence, electrical conductivity of polyaniline is reliant both on the oxidation and protonation.

As referenced previously, polyaniline is described by presence of different oxidation structures. Polyaniline as emeraldine base can be doped (protonated) to directing type of emeraldine salt. Emeraldine base, half oxidized structure, is comprised of equivalent measure of amine (-NH-) and imine (=NH-) destinations. Imine locales are exposed to protonation to shape bipolaron or dication (emeraldine salt structure). Bipolarone is additionally separated by infusion of two electrons both from electron sets of two imine nitrogen into quinodiimine ring, and the third twofold obligation of benzenoid ring is formed. Unpaired electrons at nitrogen molecules are cation extremists, yet basically, they address polarons. The polaron cross section, liable for high conductivity of polyaniline as emeraldine salt is framed by reallocation of polarons along polymer chain, as per schematic portrayal given in Fig. 2. Albeit both bipolaron and polaron hypothetical models of emeraldine salt conductivity were proposed it was recently affirmed that, adjacent to from the way that couple of cowardly bipolarons exist in polyaniline, arrangement of polarons as charge transporters clarified high conductivity of polyaniline. As referenced, exceptional property of polyaniline is conductivity reliance on the doping (proton) level. [11]

The maximal conductivity of polyaniline is accomplished at doping level of half, which compares to polyaniline as emeraldine salt. For higher doping degrees a portion of the amine destinations are protonated, while lower doping degrees implies that a portion of the imine locales were left unprotonated, clarifying why, in the light of the polaron conductivity model, decrease of emeraldine salt to leucoemeraldine and oxidation to pernigraniline states decline the conductivity. The significant degree for conductivity differs from  $10^{-2}$  S  $\text{cm}^{-1}$ , for undoped emeraldine, up to  $10^3$  S  $\text{cm}^{-1}$  for doped emeraldine salt.

## 1.5 Synthesis of conjugating polymers

Conjugated polymers are organic macromolecules with alternating single and double bonds. Owing to the delocalization of electrons along the conjugated polymer backbone, conjugated

polymers possess interesting electronic and optical properties attractive for various optoelectronic applications.

Generally speaking, there are three methods for synthesizing conjugated polymers: electrochemical polymerization, chemical oxidation polymerization, and transition-metal-catalyzed polycondensation. Among them, the transition-metal-catalyzed polycondensation method can afford the resultant conjugated.

### **1.5.1 Electrochemical polymerisation:**

The electrochemical preparation of conducting polymers is usually carried out through oxidative polymerization of their corresponding monomers by constant current, constant potential, or cyclic voltammetry in a potential range. The electro polymerization is performed in an electrolyte solution which contains solvent, electrolyte salt, and the monomer. There are many factors influencing the electro polymerization processes, such as solvents, supporting electrolyte salts, concentration of the monomers, and pH value of the electrolyte solutions, as well as polymerization potential, current, temperature, etc. Among these factors, the polymerization potential of the monomers is the most important. The lower oxidation polymerization potentials of aniline make the electro polymerization of polyaniline easier, and it can be performed in aqueous solutions. Electro polymerization is another covering method wherein the directing polymer is framed and saved from a monomer arrangement onto a leading substrate. This is normally the technique for decision in covering generally little regions.

Care should be taken in picking the electropolymerization condition, particularly the applied potential and current. The applied potential ought to be sufficiently high to oxidize the monomer and polymerize it, however low enough not to break up the metal or incite consumption. The electrochemical covering is typically done in an electrochemical cell made out of the substrate to be covered as the functioning terminal and two inactive materials as the counter cathode (normally platinum) and the reference anode (generally Ag/AgCl or SCE). The polymerization arrangement contains the monomer, dissolvable, and supporting electrolyte.

This method can be further divided into some other polymerisation

- 1 Potentiodynamic electro polymerisation
- 2 Galvanostatic electro polymerisation
- 3 Potentiostatic electro polymerisation



**Figure 1.5:** Electrochemical Polymerisation

### **1.5.1.1. Galvanostatic Electropolymerization**

Galvanostatic electropolymerization includes the use of consistent current to polymerize the directing polymer at a steady rate. Toward the start of the electropolymerization, the likely ascents for a brief period and afterward diminishes sooner or later. It has been clarified that the abrupt expansion in potential is achieved by the development of the redox-dynamic charged oligomers before the electrode. The ensuing reduction in the potential is achieved by the catalytic impact of the charged oligomers to oxidize the monomers. The calculated potential likewise relies upon the temperature—the deliberate expected reductions with an abatement in temperature. This can be clarified by the lessening of the volume of the dissolvable, which thusly expands the concentrating of the monomer as the temperature diminishes.

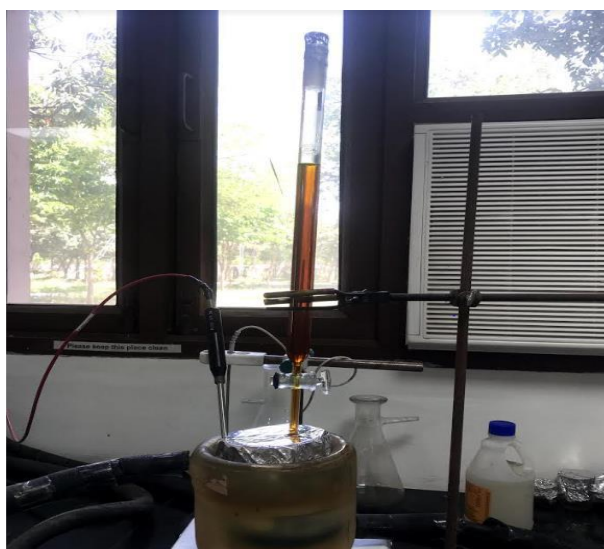
### **1.5.1.2. Potentiostatic Electropolymerization**

Potentiostatic electropolymerization includes the application steady potential. The pace of polymerization can be controlled relying upon the applied potential. This technique resembles galvanostatic electropolymerization and is unique in relation to potentiodynamic electropolymerization in light of the fact that no material is released from the saved film during the covering method. Polypyrrole films polymerized utilizing potentiostatic electropolymerization (and by galvanostatic electropolymerization) are of the dendritic kind and have low grip strength on the substrate. Interestingly, when the polypyrrole is polymerized potential powerfully, the subsequent film is glossy dark, clings firmly to the substrate, and has a smooth and homogenous surface morphology. This has been disclosed as because of the arrangement of countless comparable nucleation site during the development process.<sup>14</sup>

Poly(3-methylthiophene) films, then again, showed better electrical properties (conductivity, charge portability, number of free transporters, and band hole) when polymerized potentiostatically than when polymerized potentiodynamically.

### 1.5.2 Chemical Polymerisation

The chemical oxidation preparation of conducting polymers is performed in solution by using oxidants such as  $\text{FeCl}_3$  and  $(\text{NH}_4)_2\text{S}_{208}$ , etc., and it is easy to enlarge the production scale with the chemical polymerization. Chemical polymerization is the most important method for the preparation of PANI, and it can also be used to produce conducting PPy.



**Figure 1.6:** Chemical Polymerisation

## 1.6 Application of conjugated polymers in energy storage devices:

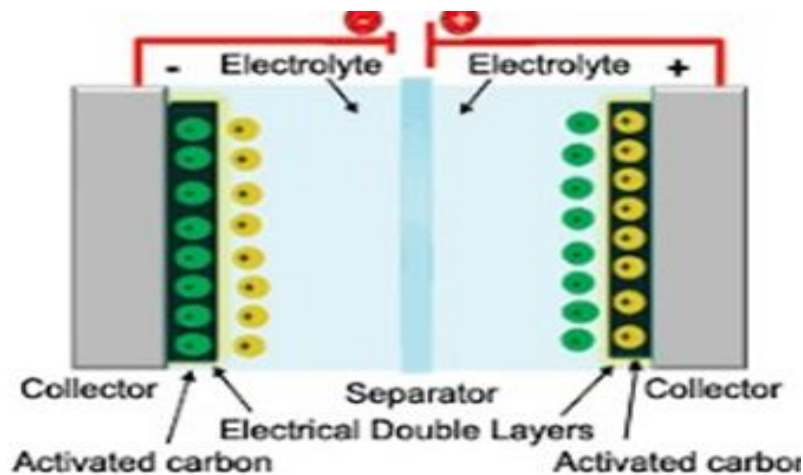
### 1.6.1 supercapacitor

A supercapacitor (SC) (likewise called a supercapacitor, ultracapacitor (or Goldcap) is a high-limit capacitor with capacitance esteems a lot higher than different capacitors (however lower voltage restricts) that overcome any barrier between electrolytic capacitors and battery-powered batteries. They ordinarily store 10 to multiple times more energy per unit volume or mass than electrolytic capacitors, can acknowledge and convey charge a lot quicker than batteries, and endure a lot more charge and release cycles than battery-powered batteries. Supercapacitors are used in applications requiring various fast charge/discharge cycles instead of long stretch insignificant energy accumulating: inside vehicles, transports, trains, cranes and lifts, where they are used for regenerative slowing down, transient energy stockpiling or burst-mode power conveyance. More modest units are utilized as memory reinforcement for static arbitrary access memory (SRAM). In contrast to normal capacitors, supercapacitors don't

utilize the ordinary strong dielectric, but instead, they utilize electrostatic twofold layer capacitance and electrochemical pseudocapacitance, the two of which add to the absolute capacitance of the capacitor, with a couple of contrasts: Electrostatic double layer capacitors (EDLCs) use carbon cathodes or subsidiaries with a lot higher electrostatic twofold layer capacitance than electrochemical pseudocapacitance, accomplishing detachment of charge in a Helmholtz twofold layer at the interface between the outer layer of a conductive terminal and an electrolyte. The partition of charge is of the request for a couple ångströms (0.3–0.8 nm), a lot more modest than in an ordinary capacitor.[6]

Electrochemical pseudocapacitors utilize metal oxide or leading polymer anodes with a high measure of electrochemical pseudocapacitance extra to the twofold layer capacitance. Pseudocapacitance is accomplished by Faradaic electron accuse move of redox responses, intercalation, or electrosorption. Crossover capacitors, for example, the lithium-particle capacitor, use cathodes with contrasting qualities: one showing generally electrostatic capacitance and the other for the most part electrochemical capacitance.

The electrolyte shapes an ionic conductive association between the two electrodes which recognizes them from customary electrolytic capacitors where a dielectric layer consistently exists, and the alleged electrolyte (e.g.,  $\text{MnO}_2$  or leading polymer) is indeed important for the subsequent terminal (the cathode, or all the more effectively the positive anode). Supercapacitors are energized by plan with asymmetric electrodes, or, for symmetric electrodes, by a potential applied during manufacture.



**Figure 1.7:** Supercapacitor

## 1.7 Preparation of GO

GO was prepared from natural graphite using the method of Hummers and Offeman.  $\text{KMnO}_4$  and  $\text{H}_2\text{SO}_4$  were used as oxidizing agents. The product was purified by washing many times with HCH (5%) and deionized water. After purification, the product was ultrasonicated for 1 h and thus a well-exfoliated GO dispersion was obtained. GO was prepared according to the modified Hummer method [18, 22]. In detail, 5 g of graphite and 2.5 g of  $\text{NaNO}_3$  were mixed with 108 mL  $\text{H}_2\text{SO}_4$  and 12 mL  $\text{H}_3\text{PO}_4$  and stirred in an ice bath for 10 min. Next, 15 g of  $\text{KMnO}_4$  were slowly added so that the temperature of the mixture remained below  $5^\circ\text{C}$ . The suspension was then responded to for 2 h in an ice shower and blended for 60 min before again being mixed in a  $40^\circ\text{C}$  water shower for 60 min. The temperature of the combination was acclimated to a steady  $98^\circ\text{C}$  for 60 min while water was added persistently. Deionized water was additionally added with the goal that the volume of the suspension was 400 mL. 15 mL of  $\text{H}_2\text{O}_2$  was added after 5 min. The response item was centrifuged and washed with deionized water and 5% HCl arrangement over and over. At last, the item was dried at  $60^\circ\text{C}$ .

## 1.8 Properties and Applications of Graphene Oxide

Due to the presence of oxygen functionalities, graphene oxide can without much of a stretch scatter in natural solvents, water, and diverse matrixes.

This is a significant advantage when joining the material with polymer or fired lattices to improve their mechanical and electrical properties. As for electrical conductivity, graphene oxide capacities as an electrical separator, in view of the aggravation of its  $\text{sp}^2$  holding organizations. Diminish the graphene oxide to recuperate the honeycomb hexagonal cross-section of graphene, to reestablish electrical conductivity. After countless oxygen bunches have been taken out, it isn't difficult to scatter the diminished graphene oxide (GO), in light of the fact that this material will in general create totals. The properties of graphene can be changed by the functionalization of graphene oxide. The artificially adjusted graphenes got by this technique might actually be utilized in a few applications. Contingent upon the expected application, the graphene oxide can be functionalized in various ways

One approach to guarantee that the artificially adjusted graphenes scatter effectively in natural solvents is to utilize amines through natural covalent functionalization, for example. This improves the material fit to creation of biodevices and optoelectronics, and for use in drug conveyance. Also, it has been shown that it is feasible to connect fullerene-functionalized



optional amines and porphyrin-functionalized essential amines to graphene oxide platelets, to improve the nonlinear optical exhibition of the material.

Graphene oxide might actually be utilized as a delegate in the creation of single-layer or not many layers graphene sheets. To accomplish this, an oxidization and decrease cycle ought to be fostered that can detach carbon layers and separate them without changing their design.

As far as the large scale manufacturing of graphene, the compound decrease of graphene oxide is viewed as quite possibly the most practical method.

However, scientists have found it challenging to create graphene sheets that have the same quality as those made by mechanical exfoliation on a large scale.[6,7]

## **1.9 Preparation of Aniline**

There are two steps to prepare aniline from benzene.

### **Benzene to nitrobenzene**

Benzene reacts with concentrated sulfuric acid and concentrated nitric acid on heating about 400C and give nitrobenzene. A -NO<sub>2</sub> group is substituted to the benzene ring.

### **Nitrobenzene to aniline**

Aniline is ready by the response of nitrobenzene and Sn/concentrated HCl/abundance NaOH with nitrobenzene. In this response, nitrobenzene is diminished to aniline salt by Sn/concentrated HCl. Then, at that point, aniline is recuperated by adding NaOH.

## **1.10 Physical properties of aniline**

- A light brown solution
- Aniline has a characteristic odor
- Aniline is a polar compound. Due to exist of hydrogen bonds, melting and boiling points are high. But H bonds of aniline is weaker than hydrogen bonds of phenol.
- Aniline can make hydrogen bonds with water. But due to large phenyl group solubility of aniline is low.
- Aniline is less basic than ammonia. But amides are less basic than aniline.

## **1.11 Pellets**

The pellets so prepared are strong and compacted enough and they are used further for the characterisations to confirm the size, shape and structure of the polymer. The pellets are formed with the different compositions having mixture of two or more compounds with different ratios.

- Go polyaniline (1:1)
- Go polyaniline (2:1)
- Go polyaniline (1:2)
- Pure polyaniline
- Pure Go

## **1.12 Characterization**

The characterisations are further explained: -

### **1.12.1 X –Ray Diffraction (XRD)**

X-ray diffraction method (XRD) is used to confirm the formation of polythiophene structure whether the formation is correct or not. These characterisations helps us to know the structure shape, size and variation towards any particular method. By this characterisation we will have the data to plot the graphs with which we can conclude the peaks for the compound.

### **1.12.2 Fourier Transformed Infrared Analysis (FTIR)**

Fourier-transform infrared analysis (FTIR) is a method used to acquire an infrared range of ingestion or outflow of a strong, fluid or gas. A FTIR spectrometer at the same time gathers high-goal ghashly information over a wide phantom reach. The term Fourier-Transform Infrared Spectroscopy begins from the way that a Fourier change (a numerical interaction) is needed to change over the crude information into the real range.

### **1.12.3 Low temperature conductivity**

The thermal (and electrical) properties of any material are identified with the vibrations of its iotas around its balance positions (in a lattice cristal). The abundancy of these vibrations relies upon temperature and reduces as the temperature diminishes. Note that these vibrations might proliferate inside the material at the speed of sound, and are examined as plane waves, with which phonons are related. thermal properties likewise rely upon the developments of negative charges (electrons) and positive charges (vacancies) if the material is a conductor.

# **Chapter -2**

## Chapter -2: Experimental details

### 2.1 Synthesis of Polyaniline Nanofibers

#### Procedure:

The chemical polymerization of aniline was carried out in the presence of aqueous sulphuric acid solution. Suitable measure of aniline (0.1 M) is taken, to which H<sub>2</sub>SO<sub>4</sub> corrosive was added into polymerization vessel and make an all-out volume of 100 ml. Then, at that point, the vessel was put on a magnetic stirrer. 20 ml of watery arrangement of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> oxidant at fitting fixation was added gradually to the response medium by drop savvy style for around 20 minutes utilizing a dropping channel.

Toward the finish of polymerization response, eventual outcome was sifted, washed with weaken arrangements of H<sub>2</sub>SO<sub>4</sub> corrosive and dried at 600C in vacuum, for 12 hours. The combined polyaniline was at long last grinded and the product is obtained in the form of powder.

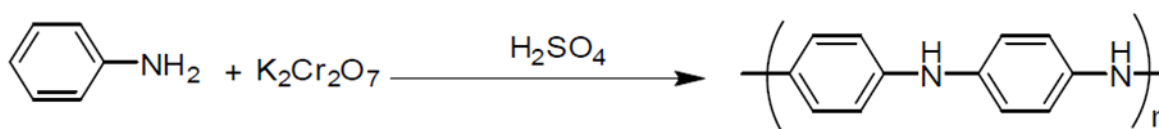
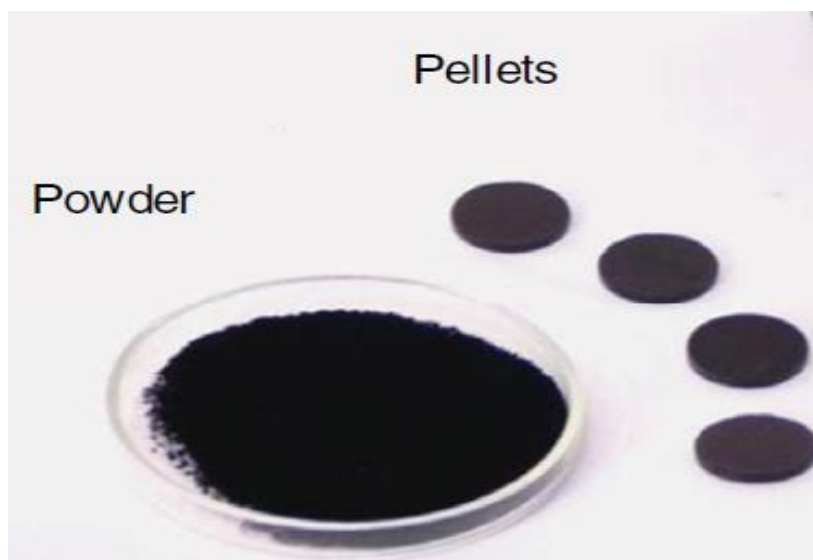


Figure2.1: Instrument Julabo



**Figure 2.2:** Obtained Polymer

## 2.2 Materials

- Round bottom flask,
- Aniline,
- Aqueous  $H_2SO_4$ ,
- Magnetic beat,
- $K_2Cr_2O_7$

## 2.3 Measurements

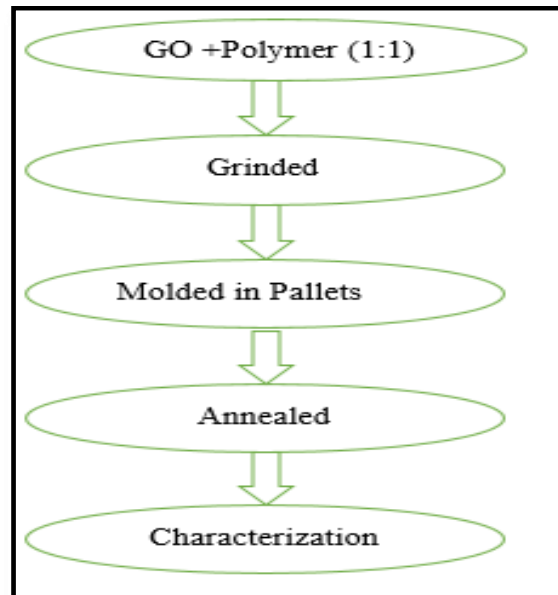
In order to determine the structure and properties of the polyaniline nanofibers, several instrumental techniques, such as XRD (XRD-Smart lab) and FTIR (Spectrum RX1), were Employed. Surface charge was determined by using zeta potential measurement (Horiba).

## 2.4 Pallet Formation

- Take a sufficient amount or compound as mentioned in the table for preparation of various pellets. Consider for the 1:1 of GO and polyaniline, measure the powdered form of polyaniline and GO and mix it with the help of compound dispenser until very small microbes formed, then add 10% of PVDF in it to bind the compound properly.
- Then with the help of spatula transfer the compound to a beaker and add few drops of chloroform in it. For mixing the compound completely use homogenator for stirring for about  $10^{-15}$  minutes. Then after stirring place the beaker having compound in the sonicator for around 30 minutes.
- After sonicating place the beaker in oven until the compound will become dry completely. When the powder form of compound is ready use die to make pellets and

use hydraulic pressure machine to give a compact form to a pellete and then to make pellete strong use annealing machine up to 150°C that provides the sufficient heat and makes the pellete stronger and compacted. And then we have a compacted and a strong pellete of 0.2 gm

- The thickness of obtained pellet is 0.005 cm and area is  $2.80 \times 10^{-5}$



**Figure2.3:** Flow chart showing Pallet Formation



**Homogenater**



**Hydraulic pressure**



**Pellets mold/die**

**Figure2.4:** Instrument Used in Pallet Formation

The table showing the composition of pallet is given below:

<b>S.NO</b>	<b>GO</b>	<b>POLYANILINE</b>	<b>PVDF</b>
1.	45%	45%	10%
2.	90%	0%	10%
3.	0%	90%	10%
4.	60%	30%	10%
5.	30%	60%	10%

**Table 1.1:** Table showing Composition of Pallet.

# Chapter -3



## Chapter -3: Characterization

In the present study, three characterisation techniques are employed which are:

- X Ray Diffraction Spectroscopy
- Fourier Transform Infrared Spectroscopy
- Low Temperature conductivity

### 3.1 X-Ray Diffraction Spectroscopy

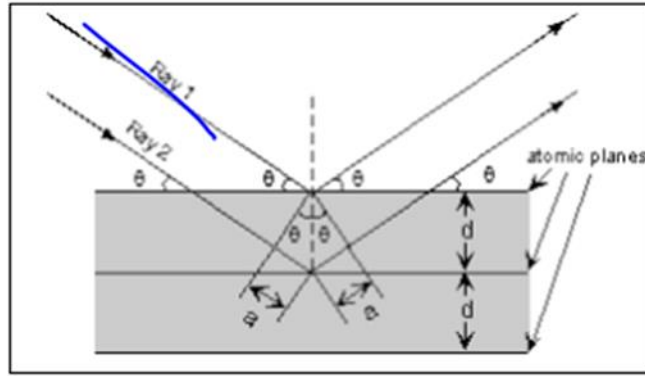
X-beam powder diffraction (XRD) is a quick insightful method principally utilized for stage distinguishing proof of a translucent material and can give data on unit cell measurements. The investigated material is finely ground, homogenized, and normal mass arrangement is resolved. Hypothesis Max von Laue, in 1912, found that translucent substances go about as three dimensional diffraction gratings for X-beam frequencies like the separating of planes in a gem cross section.

X-beam diffraction is presently a typical method for the investigation of gem structures and nuclear dividing. X-beam diffraction depends on valuable impedance of monochromatic X-beams and a translucent example. These X-beams are created by a cathode beam tube, sifted to deliver monochromatic radiation, collimated to think, and coordinated toward the example. The cooperation of the occurrence beams with the example produces useful obstruction (and a diffracted beam) when conditions fulfill Bragg's Law ( $n\lambda = 2d \sin \theta$ ).

This law relates the frequency of electromagnetic radiation to the diffraction point and the grid separating in a glasslike test. These diffracted X-beams are then recognized, handled and counted. By scanning  $2\theta$  the example through a scope of  $2\theta$  angles, all conceivable diffraction bearings of the grid ought to be achieved because of the irregular direction of the powdered material. Transformation of the diffraction tops to d-spacings permits recognizable proof of the mineral on the grounds that every mineral has a bunch of novel d-spacings.

Ordinarily, this is accomplished by examination of d-spacings with standard reference plans. points, all conceivable diffraction bearings of the cross section ought to be accomplished because of the arbitrary direction of the powdered material.

Change of the diffraction tops to d-spacings permits distinguishing proof of the mineral on the grounds that every mineral has a bunch of one-of-a-kind d-spacings. Commonly, this is accomplished by correlation of d-spacings with standard reference designs.[4]



**Figure3.1:** X-ray Diffraction Phenomenon

### 3.1.1 Instrumentation

X-ray diffractometers contains of three regular elements: a sample holder, an X-ray tube and an X-ray detector. X-Rays are created in a cathode beam tube by warming a filament to deliver electrons, speeding up the electrons toward an objective by applying a voltage, and barraging the objective material with electrons. At the point when electrons have sufficient energy to remove inward shell electrons of the objective material, characteristic X-Rays spectra are created. These spectra consist of several components, the most common being  $K\alpha$  and  $K\beta$ .  $K\alpha$  consists, in part, of  $K\alpha_1$  and  $K\alpha_2$ .  $K\alpha_1$  has a slightly shorter wavelength and twice the intensity as  $K\alpha_2$ . The specific wavelengths are characteristic of the target material (Cu, Fe, Mo, Cr).

Filtering, by foils or crystal monochrometers, is needed to create monochromatic X-Rays required for diffraction.  $K\alpha_1$  and  $K\alpha_2$  are adequately close in frequency with the end goal that a weighted normal of the two is utilized. Copper is the most widely recognized objective material for crystals diffraction, with  $2\theta$  Cu $K\alpha$  radiation =  $1.5418\text{\AA}$ . These X-beams are collimated and coordinated onto the example. As the example and finder are pivoted, the force of the reflected X-Rays is recorded. At the point when the calculation of the occurrence X-Rays impinging the example fulfills the Bragg Equation, valuable impedance happens and a top in intensity happens.

A finder records and cycles this X-Ray sign and converts the sign to a count rate which is then yield to a gadget, for example, a printer or a PC screen. The calculation of an X-Ray diffractometer is to such an extent that the example pivots in the way of the collimated X-Ray bar at a angle  $\theta$  while the X-Ray locator is mounted on an arm to gather the diffracted X-Rays and pivots at a angle of  $2\theta$  The instrument used to keep up with the point and turn the example is named a goniometer. For commonplace powder designs, information is gathered at  $2\theta$  from  $\sim 5^\circ$  to  $70^\circ$ , points that are available in the X-Ray. [4'3]

## 3.2 Fourier Transform Infrared Spectroscopy

Fourier Transform Infrared Spectroscopy Fourier transform Infrared spectroscopy (or FT-IR) is employed to find the functional groups present in an organic molecule. The instrument uses the infrared spectrum ranging from 4000 to 400  $\text{cm}^{-1}$ . It is being observed that the even a simple molecule can give complex spectrum and by matching peak by peak we can identify the no. of functional group present. Two compounds except for enantiomers cannot have same IR spectrum. IR spectrum can also be used to check the differences in the various groups formed and lost during chemical reaction by comparing with the spectrum of the reactants. Infrared frequency less than 100  $\text{cm}^{-1}$  is absorbed and converted into rotational energy in organic molecules. This is shown in the form of discrete lines as this energy is quantized.

Infrared radiation ranging from 10000-100  $\text{cm}^{-1}$  is absorbed and converted by organic molecules in the form of vibrational energy. This energy is also quantized but vibrational energy appears in the spectrum as bands rather lines because a vibrational energy change is accompanied by many rotational energy changes. We are only concerned in vibrational rotational energy belonging to the spectrum ranging between 4000 and 400  $\text{cm}^{-1}$ . The recurrence or frequency of vibration relies upon the overall masses of the particles, the force constants of the bonds, and the geometry of the atoms.

Band positions in IR spectra is defined by wavenumbers whose units is reciprocal cm. This unit is proportional to the energy of the vibration and instrument are linear in reciprocal cm. Band intensities can either be defined by transmittance or absorbance.

Transmittance is the ratio of radiant power transmitted by the sample to the radiant power incident in the sample. Absorbance is the log to the base 10 of the reciprocal of the transmittance;  $A = \log_{10}(1/T)$ .

There are two types of vibration stretching and bending, in stretching the bond length increases or decreases with constant vibrations whereas in bending the bond angle changes. Only those vibrations that results in rhythmically change in dipole movement of the molecule are observed in IR. The list of functional groups associated with the frequencies are shown in the table below.

### 3.2.1 Instrumentation

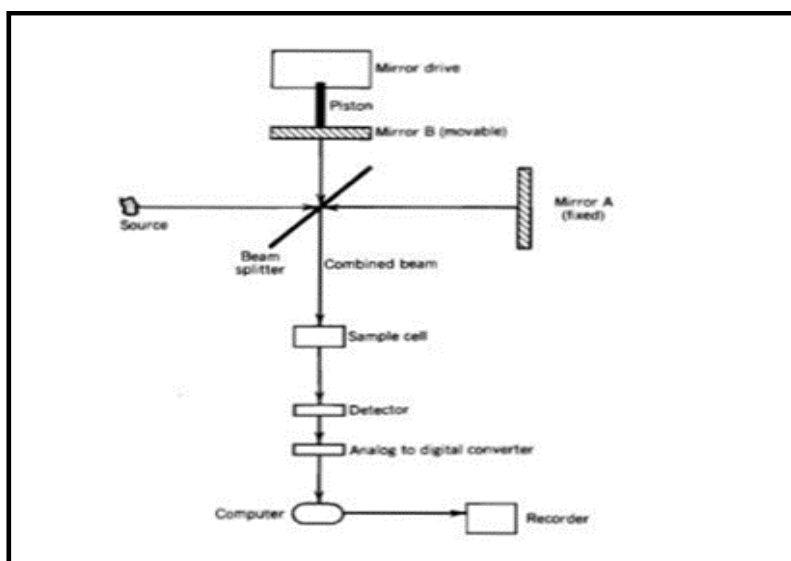
For many years, an infrared spectrum was obtained by passing an infrared beam through the sample and scanning the spectrum with diffraction grating. The spectrum was scanned by rotating the diffraction grating, the absorption areas were detected and plotted as frequency vs intensity. Over the past decade Fourier transform infrared (FT-IR) spectrometry has been

extensively developed providing several advantages. Radiation containing spectrum from 4000-400  $\text{cm}^{-1}$  is split into two beams one is of fixed length and other is of variable length. The varying distance between two way lengths bring about a grouping of helpful and dangerous impedances and subsequently varieties in intensities: an interferogram. Fourier transformation converts this interferogram from time domain into frequency domain. Smooth and continuous variation of the piston varies the path length of beam B by adjusting the position of the mirror B. Fourier transform at successive points throughout this variation gives rise to complete IR spectrum. The schematic diagram of working of FT-IR spectrometer is given in figure 2.1.

There are number of advantages to FT IR method. Since a monochromator is not used, the entire spectrum of radiation is passed through the sample simultaneously and much time is saved. FT IR have much high resolutions.

Also, since the data undergoes analogue to digital conversion it is easier to manipulate, results of several scans can be combined to average out random absorption artefacts and excellent spectra from very small samples can be obtained.

Samples can be used in solid, liquid or gases. The spectra of gases or low boiling liquids may be obtained by expansion of the sample into an evacuated cell. Gas cells are available in length of a few centimetres to 40m. Liquids can be examined neat or in solutions, neat liquids are examined between salt plates. Solid or gel form can be examined by ATR assembly which was used at NPL for FT IR characterization. Sometimes the KBr pellets are also used but the use of KBr pellets is mostly avoided because it is very less likely to make good pellets.[13]



**Figure 3.2:** Fourier Transform Infrared Spectroscopy Phenomenon

### 3.3 Low Temperature

The mechanism of polyaniline conductivity differs from other electro-conducting polymers, because nitrogen atom is involved in the formation of radical cation, unlike most of the electro-conducting polymers whose radical cation is formed at carbon. Then again, nitrogen is additionally associated with the conjugated double bonds framework. Consequently, electrical conductivity of polyaniline is reliant both on the oxidation and protonation. As referenced previously, polyaniline is described by presence of different oxidation structures. Polyaniline as emeraldine base can be doped (protonated) to directing type of emeraldine salt. Emeraldine base, half oxidized structure, is comprised of equivalent measure of amine (-NH-) and imine (=NH-) destinations. Imine locales are exposed to protonation to shape bipolaron or dication (emeraldine salt structure). Bipolarone is additionally separated by infusion of two electrons both from electron sets of two imine nitrogen, into quinodiimine ring, and the third twofold obligation of benzenoid ring is formed. Unpaired electrons at nitrogen molecules are cation extremists, yet basically, they address polarons. The polaron cross section, liable for high conductivity of polyaniline as emeraldine salt is framed by reallocation of polarons along polymer chain, as per schematic portrayal given in Fig. 2. Albeit both bipolaron and polaron hypothetical models of emeraldine salt conductivity were proposed it was recently affirmed that, adjacent to from the way that couple of cowardly bipolarons exist in polyaniline, arrangement of polarons as charge transporters clarified high conductivity of polyaniline. As referenced, exceptional property of polyaniline is conductivity reliance on the doping (proton) level. [3,10]

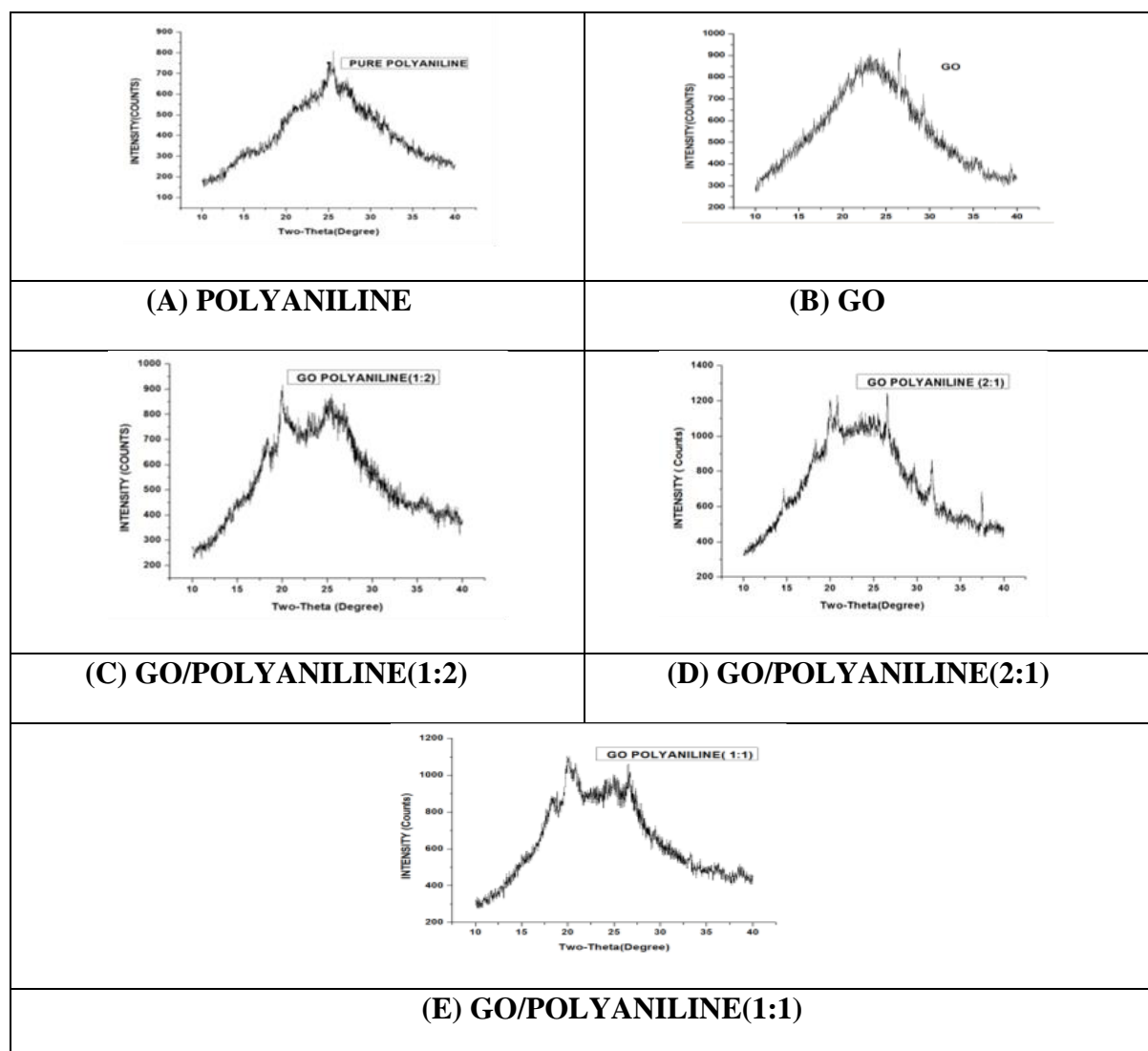
The maximal conductivity of polyaniline is accomplished at doping level of half, which compares to polyaniline as emeraldine salt. For higher doping degrees a portion of the amine destinations are protonated, while lower doping degrees implies that a portion of the imine locales were left unprotonated, clarifying why, in the light of the polaron conductivity model, decrease of emeraldine salt to leucoemeraldine and oxidation to pernigraniline states decline the conductivity. The significant degree for conductivity differs from  $10^{-2}$  S  $\text{cm}^{-1}$ , for undoped emeraldine, up to  $10^3$  S  $\text{cm}^{-1}$  for doped emeraldine salt.

# Chapter -4

## Chapter -4: Results and discussions

### (a) X- Ray Diffraction(XRD)

Crystallinity orientation of conducting polymer are of much interest, because more highly ordered systems could display a metallic property such as like conductive state. The XRD pattern for PANI doped with  $H_2SO_4$  in oxidative and electrochemical synthesis has been assessed are shown in Figure It is plainly demonstrated that the intensity of noticed peaks is better evolved on the composites arranged utilizing di and tribasic corrosive arrangements contrasted and the monobasic corrosive. The profile of the trademark pinnacle of PANI at  $2\theta \sim 25^\circ$  has the power request  $H_2SO_4 > H_3PO_4 > HCL > HNO_3$  [10, 21]. Accordingly, the small portion of the glasslike stage was observed to be expanded as expanding the voltages. The Fig. shows the oxidative blend of polyaniline, show the electrochemical blend of polyaniline. It unmistakably demonstrates that is expanded while expanding the applied voltage.



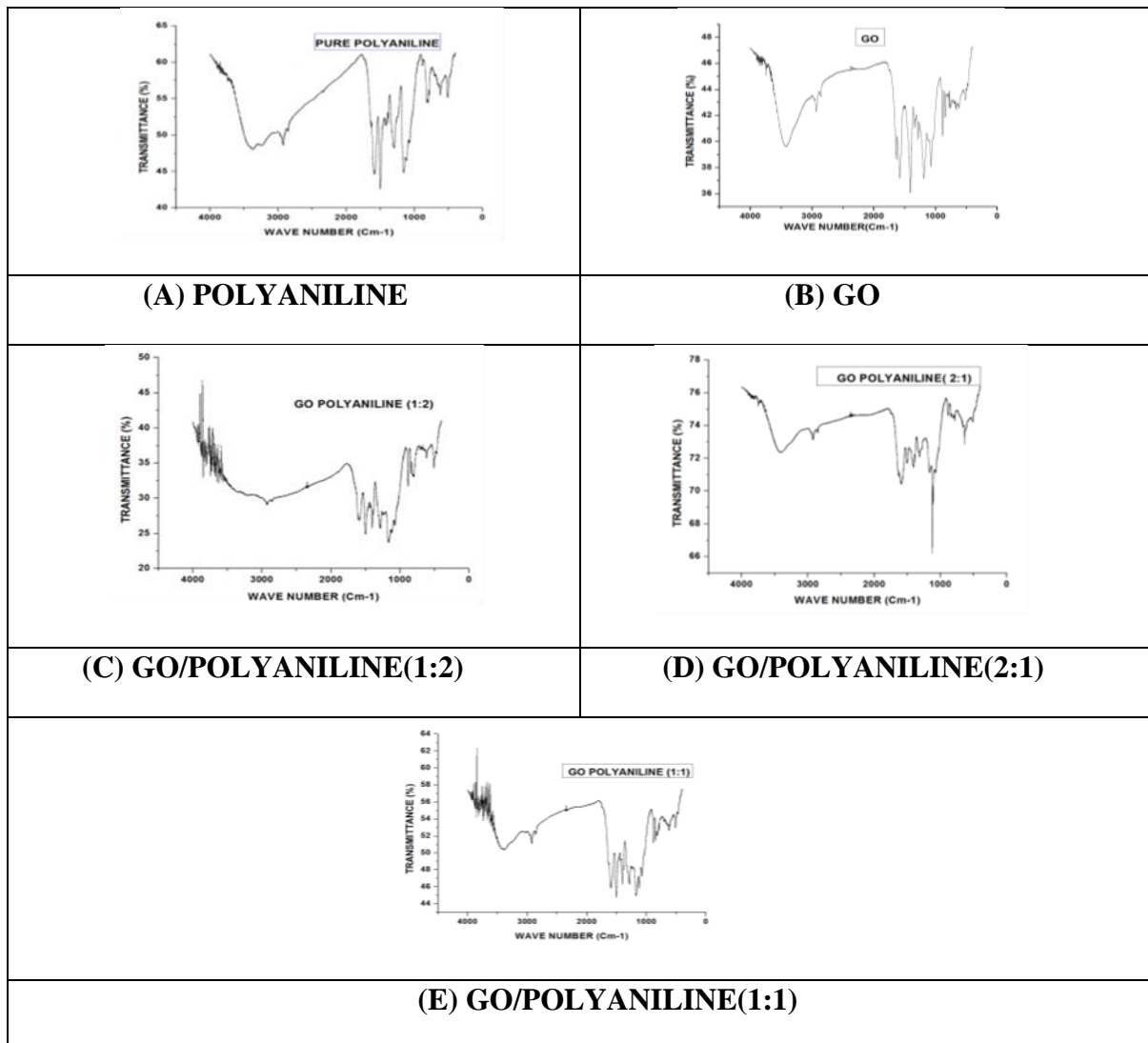
**Figure 4.1:** (A-E) Variation of Diffraction angle  $2\theta$  (degree) with change of intensity (counts)

### **(b) Fourier Transformation Infrared radiations(FT-IR)**

FT-IR spectra of PANI samples doped with H<sub>2</sub>SO<sub>4</sub> acid are shown in Fig 1. In a spectrum the band observed at 3433.75 cm<sup>-1</sup> is due to N-H stretching. The polymer shows the absorption bands at 2923.62, 2923.25, 2923.21, and 2825.55 cm<sup>-1</sup> are because of topsy-turvy C-H extending and symmetric C-H extending vibrations. The absorption tops saw at 1654.43, 1637.67, and 1637.68 cm<sup>-1</sup> were ascribed to C=C extending in aromatic nucliee.

The bands acquired at 1600-1500 cm<sup>-1</sup> relates to C-H extending in aromatic mixtures. Assimilation groups at 1476.12, 1489.67, 1490.77, and 1491.15 cm<sup>-1</sup> confirmed C=N extending in aromatic mixtures. The polymer shows assimilation bands at 1300-1200 cm<sup>-1</sup> which affirms the C-N extending of essential sweet-smelling amines. The retention bands that showed up at 1111.76, 1112.28, 1124.11, and 1137.65 cm<sup>-1</sup> uncovers the C-H bowing vibrations. The retention bands lie under 1000 cm-1 are the attributes of monosubstituted benzene. A similar finding has been accounted for somewhere else. The retention tops at 1560 cm<sup>-1</sup> allocated to the quinoide structure doesn't uncovered any critical changes for all polymer tests, that reasons that the polymers were arranged utilizing di and tri fundamental acids. It has been accounted for that H<sub>2</sub>SO<sub>4</sub> might connect with PANI by giving either hydrogen sulfate, HSO<sub>4</sub><sup>-</sup> or sulfate, SO<sub>4</sub><sup>2-</sup> anions as dopant anions. Many authors agreed that HSO<sub>4</sub><sup>-</sup> dopant anions are present in PANI/H<sub>2</sub>SO<sub>4</sub>. The figure shows the oxidative synthesis of polyaniline. Fig. shows the electrochemical amalgamation of polyaniline. The range shows some intriguing tops with regards to electrochemical strategy because of complete polymerization of customary plan. When contrasted and oxidative technique and electrochemical strategy the intensity of C=N extending of polyaniline arranged 2V and 2.5V is observed to be expanded when compared with the sample arranged at 1.5V.

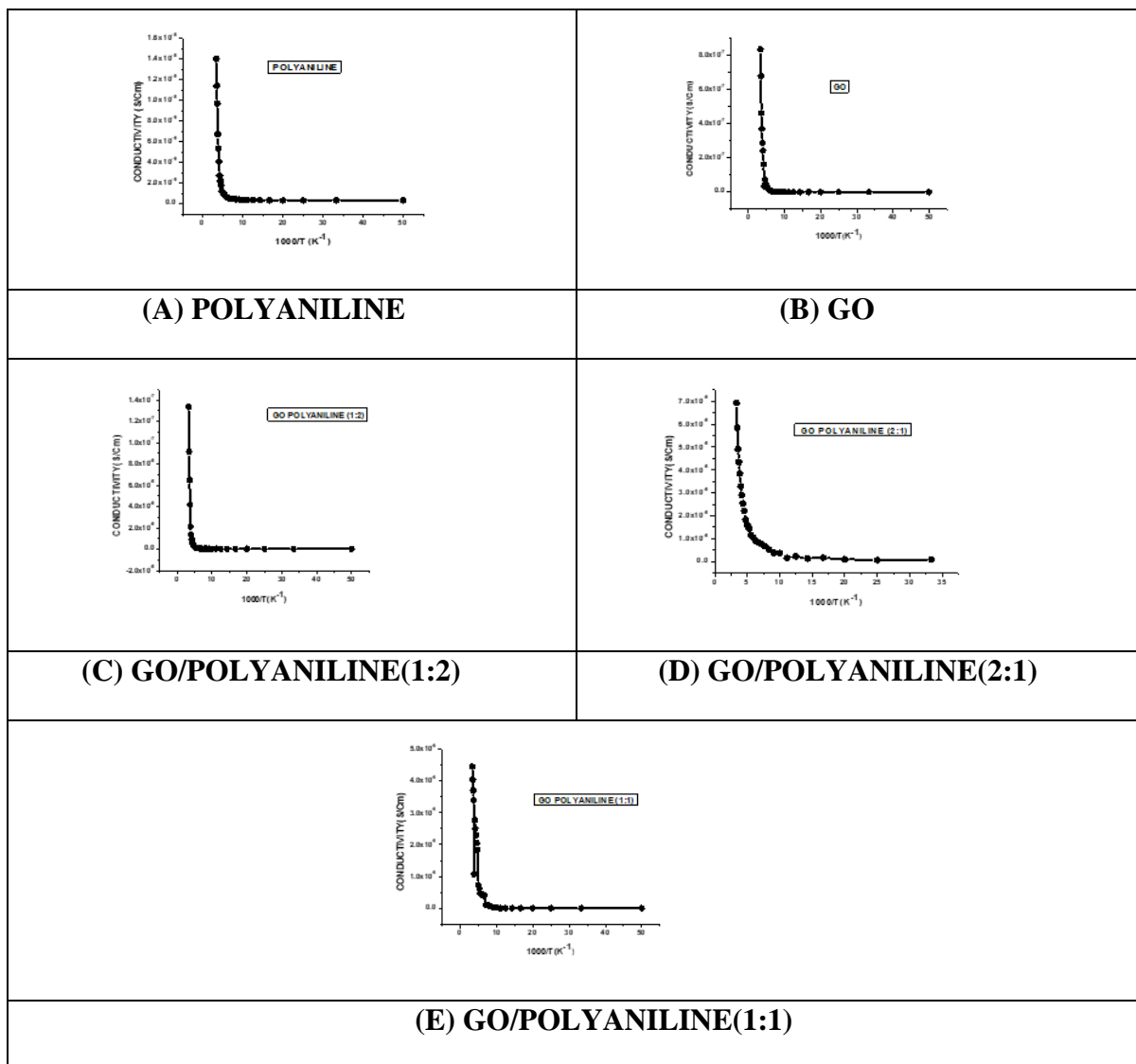




**Figure4.2:** (A-E) Variation of transmittance (%) with change of wave number

### (c) Low temperature conductivity

Graph between Conductivity & Temperature is plotted and it is concluded from the graph that the conductivity of the resulting Polyaniline/GO composite in 1:1 is  $4.45626 \times 10^{-6} \text{ S cm}^{-1}$  and 1:2 is  $1.3353 \times 10^{-7}$  and for 2:1 is  $6.9291 \times 10^{-8}$  room temperature, which is higher than that of pure polyaniline that is  $1.40177 \times 10^{-8} \text{ S/cm}$ . The DC conductivity values of the composites are seen to increase with increasing GO content. The conductivity of GO is  $8.32152 \times 10^{-7}$ . And the conductivity is decreasing with the increase in temperature.



**Figure4.3:** (A-E) Variation for Conductivity(S/Cm) with change of Temperature 1000/T

# Chapter -5

## **Chapter -5: Conclusions, applications, advantages& disadvantages**

### **5.1 Conclusions**

It is concluded from the above graph Our result shows the synthesized H<sub>2</sub>SO<sub>4</sub> polyaniline is soluble in DMSO. The conclusions arrived from the studies are as follows. The synthesized polymers were characterized by UVvisible, FTIR and XRD spectroscopic techniques. The  $\pi$ - $\pi^*$  transitions of aniline and/or anilium radicals and  $\pi$  -  $\pi^*$  advances of quinone-imine bunches were affirmed from the UV ghostly studies. The FTIR analysis affirms the compound design of polyaniline. From the X-Ray diffraction design, we can see that the crystallinity steadily increments with an increment in applied voltage. The conductivity of the electrochemical technique at 1.5V is 0.58 S/cm and in the oxidative strategy, the conductivity is 0.27 S/cm. This shows when contrasted with the oxidative strategy the electrochemical technique of synthesis is more suitable. On increasing the applied voltage, the conductivity also increases. These results reveal that they are ideally suited for the manufacture of opto- electronic devices in the visible wavelength and could be used to photo voltaic applications. Further work in this direction will be highly useful for solar cell fabrication technology.

### **5.2 Applications**

- Polyaniline and the other directing polymers, for example, polythiophene, polypyrrole, and PEDOT/PSS have a lot of potential for applications because of their light weight, conductivity, mechanical adaptability and compound properties.
- Conducting polymers polythiophene polypyrrole PEDOT/PSS Polyaniline is particularly alluring among them since it is more affordable, has three unmistakable oxidation states with various shadings and has a corrosive/base doping reaction.
- This last property makes polyaniline an optimal alternative for acid/base substance fume sensors. The various shadings, charges, and compliances of the numerous oxidation states additionally make the material exceptionally encouraging for applications like actuators, supercapacitors, and electrochromic.
- Appealing fields for current and possible usage of polyaniline is in antistatics, charge scattering or electrostatic dispersive (ESD) coatings and mixes, electromagnetic impedance safeguarding (EMI), against destructive coatings, hole injection layers, transparnt conductors, ITO substitutions, actuators, synthetic fume and arrangement based sensors, electrochromic coatings (for shading change windows, mirrors etc.), PEDOT-PSS replacements, toxic metal recovery, catalysis, fuel cells and active electronic components

such as for non-volatile memory. antistatics electromagnetic interference shielding anti-corrosive ITO actuators catalysis fuel cells non-volatile memory.

- Notwithstanding, the significant applications are in printed circuit board fabricating (last completes) and erosion insurance.

### **5.3 Advantages**

- No pollution associated with it.
- It must last for a long time.
- No maintenance cost.

### **5.4 Disadvantages**

- It has high cost of installation.
- It has low efficiency.
- During overcast day, the energy can't be delivered and furthermore around evening time we will not get solar energy.

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